

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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IN THE APPLICATION OF:

MATTHEW R. SIVIK.

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EXAMINER: AMY T. LANG

FILED: MARCH 23, 2004

GROUP ART UNIT: 1714

TITLE: FUNCTIONALISED POLYMER COMPOSITION FOR GREASE  
Wickliffe, Ohio

Declaration Under Rule 1.132

Sir,

I, Dr Gareth Fish hereby declare:

I obtained a BSc (Hons) in Chemistry in 1984 and a Ph.D. in Mechanical Engineering (Tribology) in 1990 from the Imperial College of Science, Technology and Medicine, University of London. My Ph.D. was in the field of the lubricating properties of fire resistant lubricants. From August 1990 to May 2007, I was employed by GKN plc as a grease specialist rising from senior technologist to, principal technologist. In June 1999 I was promoted global grease technology manager, initially in England and from August 2002 in Auburn Hills, Michigan. I have been employed by The Lubrizol Corporation since June 2007 as a Technology Manager for the grease group. I have a total of 26 years of experience in the field of lubricants and greases.

I confirm that I have read the application of the present invention and cited prior art references Olson (US 5,308,514) and Smith (US 4,966,722) that were listed in the office action mailed 11 May, 2010.

The examiner states the following on page 5 of the office action dated 05/11/2010:

“Additionally, as shown on page 3 of the affidavit, although the comparative grease and the three inventive greases contain the same mole equivalent of succinic acid, the compositions all differ in the amount of oil and %wt of calcium sulphate. Therefore this table is not a proper side by side comparison of inventive and comparative data.”

The examiner is respectfully requested to re-consider the interpretation of data presented by me in the declaration submitted on 18 November 2009. The following is an attempt to clarify the invention and the comparative data. The following would be known by a person skilled in the art of grease making in particular calcium sulphate grease making.

As explained in Olsen, (example 1), the key to calcium sulphate gelling is the conversion of the amorphous calcium carbonate to crystalline calcite. To do this oil, acid, water and a promoter need to be added to the highly overbased sulphate and then gelled in a closed or sealed reactor (example 1).

Traditionally, organic (C12- C40) alkylbenzene sulphonic acids are added as the acid. Olsen describes the use of a mixture of sulphonic and 12-hydroxy stearic as the acid used. In example 1, acetic acid is used as the promoter. It is also known that succinic acid can be used as the acid and that low molecular weight alcohols such as methanol and propan-2-ol can also be used as promoters.

Olsen described the 400TBN sulphate used (bottom of column 7 and top of column 8).

A similar 400 TBN sulphate is used to exemplify the present invention. The 400 TBN sulphate is characterised as follows:

42% oil

18.5% calcium C12 -- C30 alkylbenzenesulphate (0.7% calcium)

33.6% calcium carbonate (amorphous)

2.28% free lime (1.233% calcium)

Base number (TBN) = 396.5 mg KOH / g

Strong base number (DBN) = 34.7 mg KOH / g

In order for the sulphonate to gel, the micellular structure needs to be destabilized by adding the acid. Sufficient moles of acid need to be added to reduce the strong base number (DBN) down to about 15 to 20 mg KOH / g. From standard calculations, 0.14 equivalent moles of acid is needed to lower the DBN of the sulphonate from 34.7 to 15.8 mg KOH / g.

Due to succinic acid having a lower molecular weight than the C<sub>12</sub>, C<sub>20</sub> or C<sub>16-36</sub> substituted succinic acids described as inventive examples in my previous declaration of 18 November 2009, a much lower quantity of the C<sub>12</sub>, C<sub>20</sub> or C<sub>16-36</sub> substituted succinic acids is required. The reason is because in order to make the proper comparative examples of the presently claimed grease versus the grease of Olson an approximately equal number of moles of acid groups is required i.e., the acid groups of the succinic acid group. The larger number of carbon atoms in the side chain of the C<sub>12</sub>, C<sub>20</sub> or C<sub>16-36</sub> substituted succinic acids means that in order to have approximately the same mole equivalent, a higher weight of the C<sub>12</sub>, C<sub>20</sub> or C<sub>16-36</sub> substituted succinic acids is required.

Based on the remarks above, it is evident that examples G1 to G4 submitted in my previous declaration, the minor differences in the weight of oil and sulphonate ensure gelling as described above, and also balance out for the differences in the weight percentage of the various succinic acid added (whilst ensuring the mole percent is approximately equal). The only variable is the amount of succinic acid added. Table 1 of the declaration previously presented has been expanded to further explain this. Table 1 is shown below:

	Comparative Grease	Inventive Greases		
	G1	G2	G3	G4
Succinic acid carbon chain	0	12	20	16-36
Weight of 400TBN sulphonate component (g)	700	700	700	700
Weight of calcium sulphonate in component (g)	129.5	129.5	129.5	129.5
Weight of acid added (g)	16.50	40.00	55.80	57.26
Mol weight	118.1	286.4	398.6	409.0
Equivalents	0.14	0.14	0.14	0.14
Added oil (g)	350	350	350	350
Propan-2-ol promoter (g)	80	80	80	80
Water (g)	70	70	70	70
wt % oil added	33.15	32.34	31.87	31.83
wt % oil in final grease	60.82	59.70	58.64	58.56
wt % calcium sulphonate thickener in final grease	39.18	40.50	41.36	41.44

As is noted in the table above, the mole equivalents of each grease, G1 to G4 has the same mole equivalent of the named succinic acid. Therefore G1 to G4 are proper side by side comparison of inventive and comparative examples based on mole equivalents.

With lubricating greases, they are not controlled by the percentage of oil or thickener but by the penetration number as defined by ASTM method D217-02 or its international equivalent ISO 2137. After manufacturing the penetration would be determined by ASTM D217-02 and if the grease is too stiff it is cut to the correct penetration by the addition of oil. The percent weight of oil and thickener are allowed to vary, and may vary by as much as  $\pm 5$  wt % depending on how well the grease is manufactured.

With regards to penetration, it is not a precise measurement of the stiffness or consistency of grease. It is well known to persons skilled in the art of grease that there is inherent variability in the test method. According to Section 13 Precision and Bias of

ASTM D217, the repeatability (r) and reproducibility (R) are as in the following table taken directly from the standard.

Repeatability and Reproducibility

Penetration	Penetration Range	Repeatability, One Operator and Apparatus	Reproducibility, Different Operators and Apparatus
Unworked	85 to 475	8 units	19 units
Worked	130 to 475	7 units	20 units

If the unworked or worked penetrations differ by less than the repeatability they are considered to have the same consistency. This applies directly to examples G5 and G6 as presented in my previous declaration.

I further declare that all statements herein made of my own knowledge are true and all statements herein made on information and belief are believed to be true. I understand that willful false statements and the like are punishable by fine or imprisonment or both (18 U.S.C. 1001) and may jeopardize the validity of the application or any patent issuing thereon.



Dr. Gareth Fish

12. October. 2010

Date